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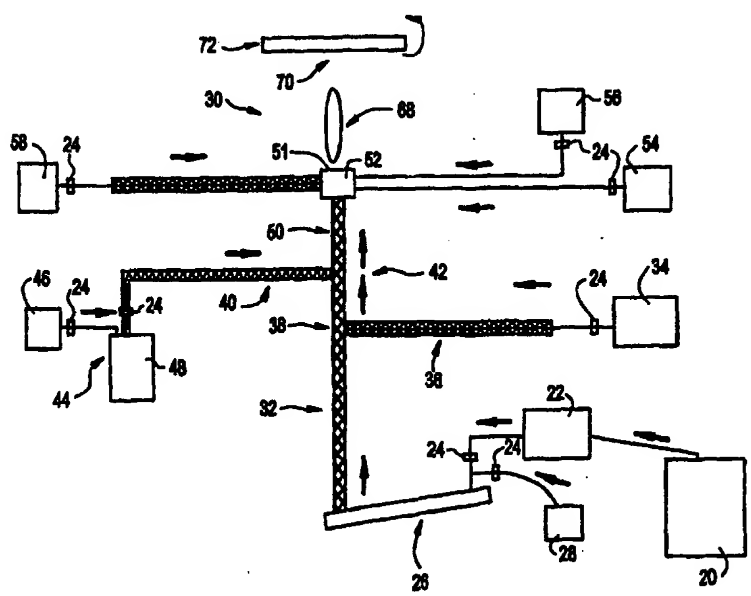

(57) Abstract

Disclosed is a method of making optical waveguide preforms and optical waveguides which may be drawn to a fiber. A silica forming feedstock which includes a mixture of siloxane and germanium chloride and an optical waveguide preform manufacturing apparatus is also disclosed. Further disclosure includes the mixing prior to deposition and use of high purity siloxane with high purity germanium chloride to produce germanium doped silica. The delivery lines for the feedstock to the deposition side may be heated to 175 °C–200 °C.

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GERMANIUM CHLORIDE AND SILOXANE FEEDSTOCK FOR FORMING SILICA GLASS AND METHOD

FIELD OF THE INVENTION

The present invention relates to silica feedstock compositions. More particularly, the present invention relates to silica forming feedstocks, and the methods of manufacturing optical waveguide preforms.

BACKGROUND OF THE INVENTION

Various processes are known that involve the production of metal oxides from a variety of feedstocks. Such processes require a feedstock and a means of catalyzing oxidation and combustion of the feedstock to convert the feedstock into finely divided aggregates called soot. This soot can be heat treated to form a high purity glass article. This process is usually carried out using specialized conversion site equipment including flame generating burners.

Historically, silicon tetrachloride has been used as the main silicon containing feedstock that is converted to silica. This use of silicon tetrachloride as the silica in forming feedstock provides a high purity silica glass and

has been the commercially preferred method of manufacturing silica glass for use in optical waveguide products and particularly the manufacturing of optical waveguide fibers and their preforms.

Organometallic siloxane compounds such as octamethylcyclotetrasiloxane have been used in the manufacture of silica glass to avoid the byproduction of HCl. U.S. Patent No. 5,296,012 by Antos et al. teaches that organometallic siloxane compounds such as octamethylcyclotetrasiloxane and chlorides such as $GeCl_4$ are chemically incompatible with each other in the vapor phase and can form particulates in the vapor transport system if mixed together before oxidation and that it is desirable to keep the vapor streams separated. U.S. Patent No. 5,296,012 discloses a complicated multiple burner method of making GeO_2 doped silica glass with separate feedstock vapor streams wherein octamethylcyclotetrasiloxane vapor is delivered to a first burner and $GeCl_4$ vapor is delivered to a separate second burner. Another prior art method kept the organometallic compound vapors separated from the dopant halide compound in separate and different delivery conduits, such as a tube with a tube, until they exited the face of a combustion burner having multiple concentric fume tubes. Such a method produced a gas stream containing the organometallic compound vapor and the dopant halide compound vapor after delivery to the conversion site combustion burner, with the compounds mixing together after exiting the conversion site combustion burner face, and just prior to entering the flame of the burner. Such prior art methods are complicated and pose production problems.

In light of this, there is a need for a germanium doped silica feedstock and a method of forming optical waveguide preforms and optical waveguide products such as

an optical fiber which avoids the problems and complications of the prior art.

SUMMARY OF THE INVENTION

Accordingly, the present invention is directed to a silica forming feedstock and method of forming optical waveguides and optical waveguide preforms that substantially obviates one or more of the problems due to limitations and disadvantages of the related art.

The principal advantage of the present invention is to provide a silica forming feedstock fluid which produces a germanium doped silica glass which allows for convenient manufacturing of optical waveguides and preforms thereof without the production of large amounts of hazardous HCl while providing the benefits of using chloride dopant precursors.

Additional features and advantages of the invention will be set forth in the description which follows, and in part will be apparent from the description, or may be learned by practice of the invention. The objectives and other advantages of the invention will be realized and attained by the methods and compositions of the invention particularly pointed out in the written description and claims hereof as well as the appended drawings.

To achieve these and other advantages in accordance with the purpose of the invention, as embodied and broadly described, the invention is a silica forming feedstock fluid including a high purity siloxane fluid and a high purity germanium chloride fluid, with the siloxane fluid preferably comprised of at least 95% by weight of octamethylcyclotetrasiloxane; preferably said germanium chloride fluid is comprised of at least 99% by weight of germanium tetrachloride. Preferably the silica forming

feedstock fluid is a vapor mixture maintained at a temperature ranging from approximately 175°C to 200°C.

In another aspect, the invention includes a method of using the inventive feedstock fluid in the formation of optical waveguide preforms and optical waveguides; preferably the method includes the steps of mixing a high purity siloxane with a high purity germanium chloride in a ratio ranging from 1-10 parts by weight of said siloxane to 1 part by weight of said germanium chloride to provide a fluid feedstock, delivering said fluid feedstock through a heated supply conduit having a temperature ranging from approximately 175°C to 200°C to a conversion site, converting said delivered fluid feedstock into germanium doped silica soot, depositing said germanium doped silica soot on a deposition surface, and forming said deposited germanium doped silica soot into an optical waveguide preform. Preferably the siloxane is comprised of at least 95% by weight of octamethylcyclotetrasiloxane. Preferably the germanium chloride is comprised of at least 99% by weight of germanium tetrachloride. Preferably the method includes the step of expelling said delivered fluid feedstock through the center tube of a conversion site burner, said center tube surrounded by an inner shield of N_2, an outer shield of O_2, and an outer ring of O_2 and fuel. Preferably the method includes the step of maintaining said fluid feedstock at a temperature ranging from approximately 190°C to 200°C prior to converting said feedstock into germanium doped silica soot.

In a further aspect, the invention includes the making of optical waveguide preforms, which are predecessors and physical embodiments of an optical waveguide product prior to the final forming of the preform into the optical waveguide product, such as by drawing a preform into an optical waveguide fiber.

In another aspect, the invention includes the forming of optical waveguide preforms by such processes as depositing, cladding, drying, consolidating, stretching, caning, overcladding, and reconsolidating.

In a further aspect, the invention includes a method of making optical fiber by converting a siloxane and germanium chloride feedstock fluid into a GeO_2 doped silica glass. The method of making an optical waveguide fiber comprises the steps of: providing a first fluid feedstock comprised of octamethylcyclotetrasiloxane and germanium tetrachloride, delivering said first fluid feedstock through a heated supply conduit having a temperature ranging from approximately 175°C to 200°C to a conversion site, converting said delivered first fluid feedstock into GeO_2 doped SiO_2 soot, depositing said GeO_2 doped SiO_2 soot on a deposition surface, providing a second fluid feedstock comprised of octamethylcyclotetrasiloxane, delivering said second fluid feedstock through a heated supply conduit having a temperature ranging from approximately 175°C to 200°C to a conversion site, converting said delivered second fluid feedstock into SiO_2 soot, depositing said SiO_2 soot over said deposited GeO_2 doped SiO_2 soot, forming said deposited SiO_2 soot and said deposited GeO_2 doped SiO_2 soot into an optical waveguide preform, and drawing said optical waveguide preform into a fiber.

In another aspect, the invention includes an optical waveguide preform manufacturing apparatus comprised of: a conversion site wherein a fluid feedstock delivered to said conversion site is converted into germanium doped silica; a means for providing a fluid feedstock comprised of a siloxane and a germanium chloride; a means for delivering said provided fluid feedstock to said conversion site; wherein said means for providing said

fluid feedstock includes a means for mixing said germanium chloride with said siloxane prior to delivering to said conversion site and said means for delivering said provided fluid feedstock includes a means for heating said fluid feedstock, preferably wherein said means for heating said fluid feedstock comprises a heated delivery conduit.

It is to be understood that both the foregoing description and the following detailed description are exemplary and explanatory and are intended to provide further explanation of the invention as claimed.

The accompanying drawings are included to provide a further understanding of the invention and are incorporated in and constitute a part of this specification, illustrate embodiments and aspects of the invention and together with the description serve to explain the principles of the invention.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 comprises a schematic representation of the method and apparatus set up of the invention.

FIG. 2 is a conversion site burner embodiment used in practicing the invention.

DETAILED DESCRIPTION OF THE INVENTION

Reference will now be made in detail to the present preferred embodiments of the invention, examples of which are illustrated in the accompanying drawings.

The silica forming feedstock fluid of the invention includes a high purity siloxane and a high purity germanium chloride. Preferably, the siloxane component of the feedstock fluid is a polyalkysiloxane, more preferably

a cyclic polyalkysiloxane, and most preferably octamethylcyclotetrasiloxane $[SiO(CH_3)_2]_4$. Preferably, the high purity siloxane is at least 95% by weight octamethylcyclotetrasiloxane, and more preferably at least 98% by weight octamethylcyclotetrasiloxane, and most preferably at least 99% by weight octamethylcyclotetrasiloxane. Preferably, the high purity germanium chloride is germanium tetrachloride ($GeCl_4$). Other germanium chlorides, such as $R_{4-n}GeCl_n$ (where n=1,2,3), organo germanium chlorides (organochlorogermanes), and chlorinated germanium compounds may be used as alternatives to the preferred $GeCl_4$. Trimethylgermanium chloride $[(CH_3)_3ClGe]$ and Methyltrichlorogermane (CH_3Cl_3Ge) are examples of such chlorinated germanium compounds. Methyltrichlorogermane (CH_3Cl_3Ge), Dimethyldichlorogermane ($(CH_3)_2Cl_2Ge$), Allyltrichlorogermane ($C_3H_5Cl_3Ge$), Phenyltrichlorogermane ($C_7H_9Cl_3Ge$) are examples of such organo germanium chlorides with the general form of R_nGeCl_{4-n} (where n=1 to 3 and R= alkyl, aryl, or alkenyl group or any combination of these groups).

Preferably, the high purity germanium chloride is at least 99% by weight germanium tetrachloride, and more preferably contains less than 10 ppb (parts per billion) each of Al, Co, Cr, Cu, Fe, Mn, Mo, Ni, Ti, V and a combined maximum total of these less than 25 ppb, and less than 5 ppm (parts per million) OH, less than 1 ppm CH, and less than 1 ppm HCl.

The preferred silica forming feedstock fluid of the invention is a vapor mixture of octamethylcyclotetrasiloxane and germanium chloride, with a further preferred vapor mixture including oxygen to improve the conversion of the feedstock into GeO_2 doped SiO_2 soot at the conversion site burner flame. The silica forming feedstock fluid vapor mixture of octamethylcyclotetra-

siloxane and germanium chloride should be maintained at a temperature of at least 175°C, preferably at least 185°C, and more preferably at least 190°C. Preferably this vapor mixture should be maintained at a temperature no greater than approximately 200°C. These elevated temperatures provide for the silica forming feedstock fluid vapor mixture to remain in the vapor state for efficient delivery to the conversion site burner without complications. It is preferred that the temperature not be too high in order to avoid any adverse reactions prior to reaching the conversion site flame.

To produce a silica soot and silica glass doped only with GeO_2, the silica forming feedstock fluid consists essentially of a siloxane and germanium chloride, preferably with octamethylcyclotetrasiloxane as the siloxane and germanium tetrachloride as the germanium chloride. To produce an optical waveguide silica glass consisting of only SiO_2 and GeO_2, the optical waveguide silica feedstock fluid consists of octamethylcylotetra-siloxane and germanium chloride.

An exemplary schematic representation of the method of making an optical waveguide preform utilizing the inventive fluid feedstock is shown in FIG. 1.

The inventive method of making an optical waveguide preform includes the steps of providing a fluid, preferably vapor, feedstock comprised of a high purity siloxane and a high purity germanium chloride and delivering the fluid feedstock through a heated supply conduit having a temperature ranging from about 175°C to 250°C, to maintain the feedstock temperature in this range, to a conversion site. As shown in FIG. 1, a siloxane liquid which comprises the siloxane component of the feedstock is contained in siloxane liquid container 20. Controllable pump 22 delivers siloxane liquid through

conduits and valve 24 to siloxane vaporizer 26. Means 28 for supplying a controllable flow of N_2 (nitrogen) carrier gas provides nitrogen carrier gas which aids in the vaporization of the siloxane liquid and delivery of the siloxane vapor to conversion site 30, which preferably is comprised of a burner and a conversion flame. Siloxane vapors are delivered through heated siloxane vapor conduit 32, which is preferably stainless steel tubing heated and maintained at 190°C. Prior to conversion site 30, oxygen is added to the siloxane vapor to assist the conversion of the fluid feedstock. Preferably oxygen supply means 34, supplies a controllable amount of oxygen vapors which are heated to an elevated temperature, preferably about 200°C. Heated oxygen from conduit 36 is added to the siloxane vapor flowing to conversion site 30 at oxygen addition junction 38.

Germanium chloride vapors from germanium chloride vapor conduit 40 are mixed with the siloxane vapors at germanium chloride addition junction 42. The mixing of germanium chloride vapors with siloxane vapors provides the homogeneous fluid feedstock comprised of siloxane and germanium chloride. Germanium chloride vapors are provided through conduit 40 by a means for controllably supplying germanium chloride such as germanium chloride vaporizer 44 which comprises a heated container of liquid germanium chloride 48 and an oxygen supply 46 wherein oxygen is bubbled through the liquid germanium chloride to assist in the formation of germanium chloride vapors. Valves 24 provide a means for controlling the amount of germanium chloride delivered through conduit 40 and mixed with siloxane vapors at junction 42. Preferably the liquid germanium chloride in container 48 is heated to 45°C and germanium chloride vapor conduit 40 is maintained at 190°C. The mixture of siloxane vapor and germanium chloride vapor fluid feedstock is provided and delivered

through heated burner supply conduit 50, which is heated to 190°C, to the single fume tube of burner 52, along with the oxygen and nitrogen gases.

Burner face 51 of burner 52 is shown in FIG. 2. Conversion site 30 includes conversion site flame 68 produced by conversion site burner 52.

The fluid vapor feedstock of siloxane and germanium chloride vapors delivered to conversion site 30 is converted by conversion site flame 68 into germanium (GeO_2) doped silica (SiO_2) soot. The germanium doped silica soot is then deposited on deposition surface 70. The deposited germanium doped silica soot is collected on deposition surface 70 which may comprise a rotating bait rod. The deposited doped soot forms the core of an optical waveguide preform. Forming the deposited germanium doped silica soot includes the steps of overcladding the germanium doped silica soot core preform with silica soot, removing the bait rod, and consolidating the porous soot preform into a nonporous soot preform.

The step of providing a fluid feedstock comprised of a high purity siloxane and a high purity germanium chloride preferably comprises the step of providing a fluid feedstock of octamethylcyclotetrasiloxane and germanium tetrachloride, and more preferably comprises the step of mixing vapors of octamethylcyclotetrasiloxane and germanium chloride prior to being delivered to conversion site 30 and burner 52. Preferably oxygen is mixed with the vapors of octamethylcyclotetrasiloxane and germanium chloride. The steps of providing and delivering a fluid feedstock of siloxane and germanium chloride preferably include the step of maintaining the fluid feedstock at a temperature range of about 175°C to 200°C, more preferably 190°C to 200°C, preferably by heating the feedstock delivery supply conduits.

The step of forming the deposited germanium doped silica soot into an optical waveguide preform includes the step of cladding the germanium doped silica soot with silica (SiO_2) soot. Preferably the step of cladding with silica soot includes the steps of providing a fluid feedstock of siloxane, delivering the fluid siloxane feedstock to conversion site 30 through heated supply conduits, converting the delivered fluid siloxane feedstock into silica soot, and depositing the silica soot on top of the germanium doped silica soot.

The invention includes the method of making an optical waveguide fiber which includes the steps of providing a fluid feedstock comprised of siloxane and germanium chloride, delivering the fluid feedstock to a conversion site, converting the delivered fluid feedstock into germanium (GeO_2) doped silica (SiO_2) soot, depositing the GeO_2 doped SiO_2 soot on a deposition surface, forming the deposited GeO_2 doped SiO_2 soot into an optical waveguide preform, and drawing the optical waveguide preform into a fiber.

The fluid feedstock of siloxane and germanium chloride, which may further include oxygen and the nitrogen (N_2) carrier gas, is delivered through heated delivery conduit 50 to the center fume tube 60 of burner 52. As shown in FIG. 2, the fluid feedstock of siloxane and germanium chloride is delivered through center fume tube 60 to conversion site flame 68. Conversion site flame 68 and the conversion of the fluid feedstock into GeO_2 doped SiO_2 soot is maintained by delivering nitrogen (N_2) gas to inner shield 62, oxygen (O_2) gas to outer shield 64, and a premixture of O_2 and fuel, preferably CH_4, to fuel-oxygen outer ring 66. Inner shield N_2 supply 58 provides N_2 gas, preferably heated and maintained at about 200°C, to nitrogen inner shield 62. Outer shield O_2 supply 54 provides O_2 gas to oxygen outer shield 64. Premix fuel-

oxygen supply 56 provides a mixture of oxygen and fuel, preferably CH_4, to fuel-oxygen outer ring 66. This provides for a beneficial conversion of the siloxane and germanium feedstock into GeO_2 doped SiO_2 soot.

The method of the invention includes the step of depositing the GeO_2 doped SiO_2 soot on a deposition surface. The GeO_2 doped SiO_2 soot is deposited and collected on deposition surface 70 of bait rod 72 to form the preform of an optical waveguide core. When a sufficient amount of GeO_2 doped SiO_2 soot is deposited on the deposition surface to form an optical waveguide core, the delivery of the germanium chloride and siloxane fluid feedstock mixture to burner 52 is halted.

The method of the invention further includes the step of forming the deposited GeO_2 doped SiO_2 soot into an optical waveguide preform. Siloxane fluid 20 can be delivered to burner 52 in place of the feedstock mixture of siloxane and germanium chloride in order to form a cladding over the deposited GeO_2 doped SiO_2 soot. Siloxane delivery can operate in the same manner as the germanium chloride and siloxane vapor feedstock mixture delivery system but only deliver siloxane, preferably octamethylcyclotetrasiloxane, O_2, and N_2 to burner 52 which is converted at conversion site 30 by flame 68 into undoped SiO_2 soot. This undoped SiO_2 soot is deposited over the GeO_2 doped SiO_2 soot to form the preform of the optical waveguide cladding.

After a sufficient amount of undoped SiO_2 soot is deposited over the GeO_2 doped SiO_2 soot, the deposition of soot is halted. The porous soot optical waveguide preform which has formed around the bait rod is removed from the bait rod. The porous soot preform is dried in a helium and chlorine atmosphere and sintered into a clear, fully dense consolidated glass cylindrical optical waveguide preform that is comprised of a GeO_2 doped silica

waveguiding core structure surrounded by a silica cladding structure. This consolidated preform is stretched into an optical waveguide cane preform. This preform is overcladded with additional undoped silica soot such as produced during the formation of the cladding soot.

The overcladded preform is reconsolidated and may be drawn into an optical waveguide fiber.

Example

The system and apparatus of FIG. 1 and 2 were used to produce GeO_2 doped SiO_2 soot in accordance with the following table. High purity liquid octamethylcyclotetrasiloxane, which was comprised of at least 95% by weight of octamethylcyclotetrasiloxane was pumped from container 20 to inclined plane flash vaporizer 26 which was heated above the boiling point of octamethylcyclotetrasiloxane. The octamethylcyclotetrasiloxane vapors were delivered towards conversion site 30 by the nitrogen carrier gas from N_2 supply 28. Delivery supply conduits 32, 50, and 40, and delivery conduit junctions 38 and 42 comprised stainless steel tubing that was heated to 190°C. Fume O_2 which was heated to about 200°C was supplied at junction 38 by O_2 source 34 and heated conduit 36. High purity $GeCl_4$ vapor was mixed with the octamethylcyclotetrasiloxane vapor at delivery junction 42. $GeCl_4$ vapors were supplied by $GeCl_4$ vaporizer 44. $GeCl_4$ vaporizer 44 comprised a $GeCl_4$ bubbler. High purity liquid $GeCl_4$ comprised of at least 99% by weight of $GeCl_4$ contained in container 48 was heated to 45°C and O_2 from O_2 source 46 was bubbled through the heated liquid $GeCl_4$. The amount of $GeCl_4$ vapor produced could be controlled by changing the flow rate of O_2 through the bubbler. This fluid feedstock vapor mixture was delivered to conversion site flame 68 through center

fume tube 60 of burner 52. This delivered fluid feedstock was converted into GeO_2 doped SiO_2 soot by conversion site flame 68 and deposited on deposition surface 70. GeO_2 doped SiO_2 soot was produced using the following conditions table:

TABLE FOR MAKING GeO_2 DOPED SiO_2 SOOT

Example #	1	2	3	4	5
Octamethylcyclotetrasiloxane [22] liquid delivery rate (grams/min.)	6	6	6	8	10
Carrier N_2 rate (liters/min.) [28]	1.25	1.25	1.25	1.25	1.25
Fume O_2 rate (liters/min.) [34]	1.8	1.8	1.8	2.5	2.5
Bubbler O_2 rate (liters/min.) for $GeCl_4$ vaporizer [44]	0.3	0.6	1.0	0.6	1.0
Inner Shield N_2 rate (liters/min.) [58]	3	3	3	3	3
Outer Shield O_2 (liters/min.) [54]	6.8	6.8	6.8	8	8
CH_4 rate (liters/min.) for premix fuel-oxygen [56]	1.1	1.1	1.1	1.1	1.1
O_2 rate (liters/min.) for premix fuel-oxygen [56]	0.9	0.9	0.9	0.9	0.9
Wt.% of GeO_2 in the SiO_2 soot	14.2%	23.0%	35.3%	7.1%	9.9%

It was surprising and unexpected that this method and use of the germanium chloride octamethylcyclotetrasiloxane fluid silica forming feedstock performed so well in the manufacturing of silica soot without evidence of adverse reaction between the octamethylcyclotetrasiloxane and germanium tetrachloride in delivery conduit 50, junction 42, or burner 52. No build up of unwanted soot or other troublesome byproducts were formed in burner 52 or on burner face 51 during the making of GeO_2 doped SiO_2 soot. After more than 16 hours of operation no build-up, corrosion, gels or other troublesome deposits were found in junction 42, conduit 50, or burner 52. This method was able to produce germanium doped silica soot doped with about 7% to 36% GeO_2 by weight. The preferred weight ratio of octamethylcyclotetrasiloxane to germanium tetrachloride of the feedstock is in the range of 1.5-7.5 parts of octamethylcyclotetrasiloxane to 1 part germanium tetrachloride, and more preferred 1.9-3.6 parts of octamethylcyclotetrasiloxane to 1 part germanium tetrachloride. The preferred delivery rates to provide the fluid feedstock is about 6-10 grams/min. octamethylcyclotetrasiloxane and 0.8-4.2 grams/min. $GeCl_4$.

It will be apparent to those skilled in the art that various modifications and variations can be made in the present invention without departing from the spirit or scope of the invention. Thus, it is intended that the present invention cover the modifications and variations of this invention provided they come within the scope of the appended claims and their equivalents.

What is claimed is:

1. A method of making an optical waveguide preform comprising the steps of:

(a) mixing a high purity siloxane with a high purity germanium chloride in a ratio ranging from 1-10 parts by weight of said siloxane to 1 part by weight of said germanium chloride to provide a fluid feedstock;

(b) delivering said fluid feedstock through a heated supply conduit having a temperature ranging from approximately 175°C to 200°C to a conversion site;

(c) converting said delivered fluid feedstock into a germanium doped silica;

(d) depositing a germanium doped silica on a deposition surface; and

(e) forming said deposited germanium doped silica into an optical waveguide preform.

2. A method as claimed in claim 1, wherein said siloxane is comprised of at least 95% by weight of octamethylcyclotetrasiloxane.

3. A method as claimed in claim 1, wherein said germanium chloride is comprised of at least 99% by weight of germanium tetrachloride.

4. A method as claimed in claim 1 further comprising the step of expelling said delivered fluid feedstock through a

center tube of a conversion site burner, said center tube surrounded by an inner shield of N_2, an outer shield of O_2, and an outer ring of O_2 and fuel.

5. A method as claimed in claim 2, wherein the step of mixing octamethylcyclotetrasiloxane and germanium chloride further comprises the step of mixing vapors of octamethylcyclotetrasiloxane and germanium chloride prior to delivering said fluid feedstock to said conversion site.

6. A method as claimed in claim 5, wherein the step of mixing vapors of octamethylcyclotetrasiloxane and germanium chloride further comprises the step of mixing vapors of octamethylcyclotetrasiloxane, germanium chloride, nitrogen and oxygen.

7. A method as claimed in claim 1, further comprising the steps of maintaining said fluid feedstock at a temperature ranging from approximately 190°C to 200°C prior to converting said feedstock into germanium doped silica.

8. A method as claimed in claim 1, wherein the step of forming said deposited germanium doped silica into an optical waveguide preform further comprises the step of cladding said germanium doped silica with silica.

9. A method as claimed in claim 8, wherein the step of cladding said germanium doped silica with silica further comprises the steps of providing a fluid feedstock comprised of a siloxane, delivering said fluid siloxane feedstock to a conversion site, and converting said delivered fluid siloxane feedstock into SiO_2.

10. A method of making an optical waveguide fiber comprising the steps of:

(a) providing a first fluid feedstock comprised of octamethylcyclotetrasiloxane and germanium tetrachloride;

(b) delivering said first fluid feedstock through a heated supply conduit having a temperature ranging from approximately 175°C to 200°C to a conversion site;

(c) converting said delivered first fluid feedstock into GeO_2 doped SiO_2 soot;

(d) depositing said GeO_2 doped SiO_2 soot on a deposition surface;

(e) providing a second fluid feedstock comprised of octamethylcyclotetrasiloxane;

(f) delivering said second fluid feedstock through a heated supply conduit having a temperature ranging from approximately 175°C to 200°C to a conversion site;

(g) converting said delivered second fluid feedstock into SiO_2 soot;

(h) depositing said SiO_2 soot over said deposited GeO_2 doped SiO_2 soot;

(i) forming said deposited SiO_2 soot and said deposited GeO_2 doped SiO_2 soot into an optical waveguide preform; and

(j) drawing said optical waveguide preform into a fiber.

11. A silica forming feedstock fluid comprised of a high purity siloxane fluid and a high purity germanium chloride fluid, said siloxane fluid comprised of at least 95% by weight of octamethylcyclotetrasiloxane.

12. A silica forming feedstock fluid as claimed in claim 11 wherein said germanium chloride fluid is comprised of at least 99% by weight of germanium tetrachloride.

13. A silica forming feedstock fluid as claimed in claim 11 comprised of a vapor mixture of octamethylcyclotetrasiloxane and germanium chloride maintained at a temperature ranging from approximately 175°C to 200°C.

14. A silica forming feedstock fluid consisting essentially of a siloxane and germanium chloride.

15. A silica forming feedstock fluid as claimed in claim 14 consisting essentially of octamethylcyclotetrasiloxane and germanium chloride.

16. An optical waveguide silica feedstock fluid consisting of octamethylcyclotetrasiloxane and germanium chloride.

17. An optical waveguide preform manufacturing apparatus comprised of:

a conversion site wherein a fluid feedstock delivered to said conversion site is converted into germanium doped silica;

a means for providing a fluid feedstock comprised of a siloxane and a germanium chloride;

a means for delivering said provided fluid feedstock to said conversion site;

wherein said means for providing said fluid feedstock includes a means for mixing said germanium chloride with said siloxane prior to delivering to said conversion site and said means for delivering said provided fluid feedstock includes a means for heating said fluid feedstock.

18. The apparatus of claim 17, wherein said means for heating said fluid feedstock comprises a heated delivery conduit.

FIG. 1

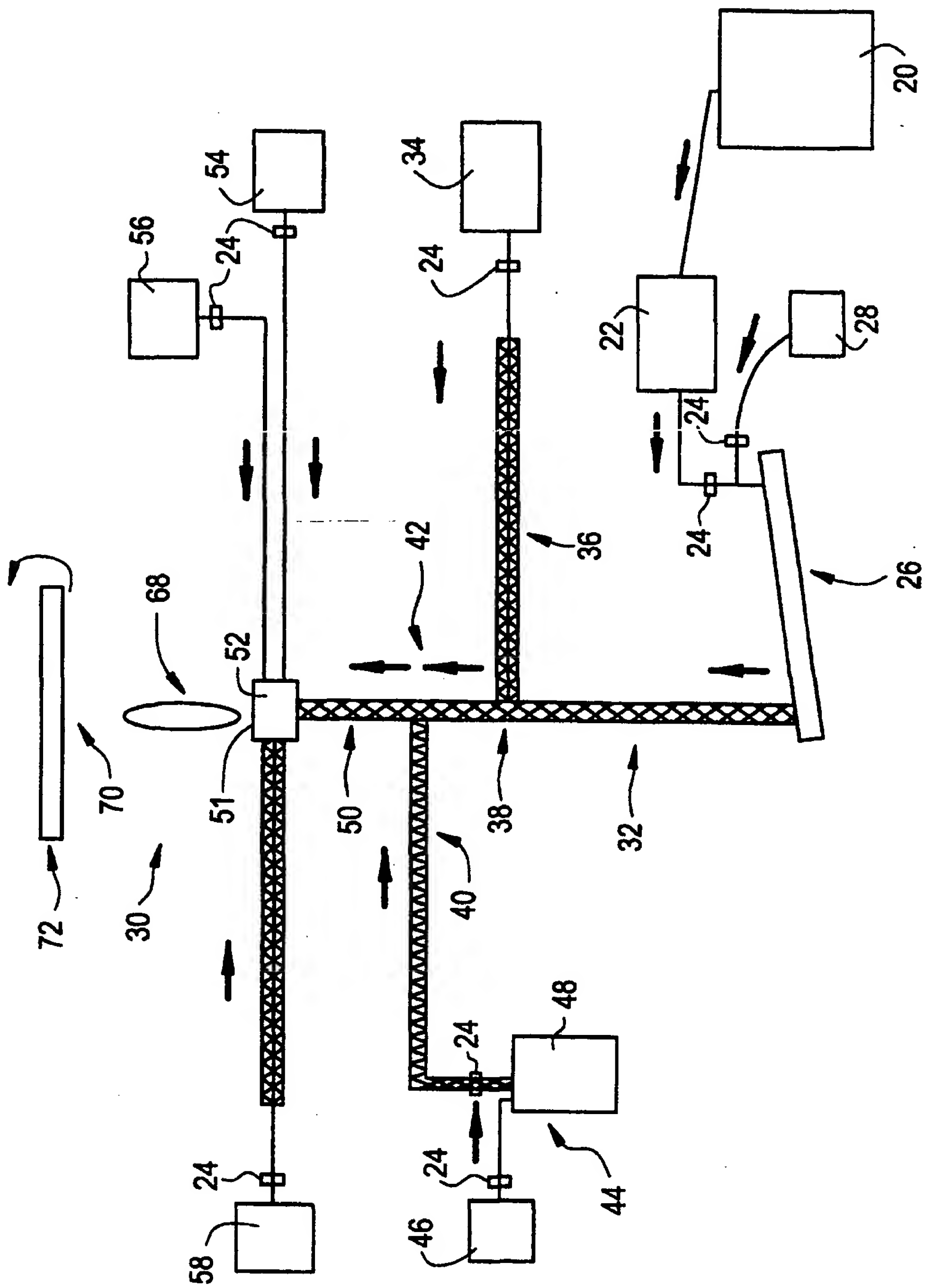
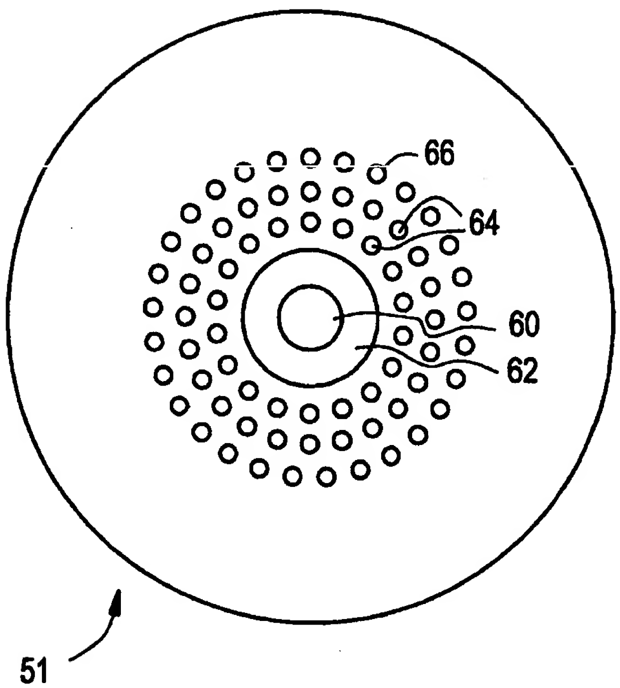


FIG. 2



INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 98/13401

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C03B37/014 C03B19/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C03B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 296 012 A (A.J.ANTOS ET AL.) 22 March 1994 cited in the application see column 4, line 47 - line 63	1,10,11, 14,16,17
A	US 5 043 002 A (M.S.DOBBINS ET AL.) 27 August 1991 see claims 7-12	1,10,11, 14,16,17
A	EP 0 529 189 A (CORNING INC.) 3 March 1993 see page 5, line 30; claims 3,5	1,10,11, 14,16,17
A	EP 0 146 659 A (SHIN-ETSU CHEM. CO.,LTD.) 3 July 1985 see examples 8-10; table 3	1,10,11, 14,16,17

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
5 October 1998	16/10/1998

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Stroud, J

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
A	PATENT ABSTRACTS OF JAPAN vol. 17, no. 205 (C-1051), 22 April 1993 & JP 04 349147 A (FUJIKURA LTD.), 3 December 1992 see abstract ---	1,10,11, 14,16
A	WO 97 22553 A (CORNING INC.) 26 June 1997 see page 7, line 27 - page 8, line 7; claims 8,9,12 see page 13, line 16 - line 26 ---	1,10,17
A	WO 90 10596 A (TSL GROUP) 20 September 1990 see page 13 - page 14 ---	1,10,11, 14,16,17
X	US 5 141 549 A (R.P.TUMMINELLI) 25 August 1992 see the whole document ---	17,18
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